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Thirtieth day of April 2004

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PROVISIONAL SPECIFICATION

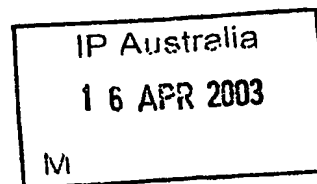
Applicant(s):

WMC RESOURCES LTD

Invention Title:

A SOLVENT EXTRACTION PROCESS

The invention is described in the following statement:



A SOLVENT EXTRACTION PROCESS

The present invention relates to the use of conductivity modifiers in a solvent extraction process, in particular a solvent extraction process for extracting copper from an aqueous medium.

Large industrial processing facilities, for example solvent extraction plants, can be quite hazardous due to their size and complexity and the nature of the materials used in the plants. Fire is a typical hazard and the fire-safety levels of a plant can vary quite dramatically as a result of even a small change at any one or more stages in a process. A small change can also have unpredictable consequences downstream. These factors make it quite difficult to ensure fire safety is adequate at all stages in a large processing plant. Also, there can be many potential causes of fire and merely recognizing one or more of these are a problem of itself.

In basic terms, a solvent extraction process as the term is used herein is a process in which an aqueous medium containing one or more metals in solution is brought into contact with organic solvent containing a dissolved extractant to produce an emulsion. After extraction of a specific metal from the aqueous medium into the solvent phase has taken place, the aqueous and solvent phases are separated using large settler tanks. Typically, solvent extraction plants include long runs of pipe work that carry a range of liquids including organic solvent, solvent containing extractant, and aqueous solutions. This range of liquids in long runs of pipe work is difficult to monitor to recognise any change which is likely to increase the potential for a fire.

The present invention is based on the realisation that a build-up and discharge of static electricity in a

solvent extraction process is one cause of fires in solvent extraction plants operating with solvents at temperatures well below the flashpoints of the solvents.

5 The present invention is also based on the realisation that it is possible to minimise the build-up and discharge of static electricity by adding conductivity modifiers to the liquids without adversely affecting the performance of the solvent extraction process.

10 Accordingly, in broad terms, the present invention provides a solvent extraction process that includes operating the process using an organic solvent that contains an amount of a conductivity modifier that
15 increases the electrical conductivity of the solvent to reduce build-up of static electricity in the process and thereby reduce the electrostatic discharge hazard of the solvent to an adequate fire safety level.

20 In addition, in broad terms, the present invention provides an organic solvent that includes a conductivity modifier for use in the above described solvent extraction process.

25 The term "conductivity modifier" is understood herein to mean a reagent that can modify the conductivity of a solvent.

30 The present invention was made during the course of an on-going research program on a copper solvent extraction plant that operates using a narrow-cut kerosene as the solvent at the Olympic Dam mine of the applicant. The research program has included laboratory bench trials and a mini-plant continuous trial.

35 The term "narrow-cut kerosene" is understood herein to mean a petroleum-derived hydrocarbon solvent

containing a mixture of aliphatic and aromatic hydrocarbons typically in the range of C10-C12.

5 Narrow-cut kerosene is flammable in the range
0.7 to 6.0% by volume with air, has a relatively high flashpoint (typically, above 75°C), and a relatively high boiling point (typically, above 195°C).

10 Kerosene is a common solvent, which is stable
under normal use conditions and is used in a variety of domestic and industrial applications. These applications range from small lamps and heaters through to large-scale mining processes. Due to its relatively high flashpoint, narrow-cut kerosene is defined as a combustible solvent
15 rather than a flammable solvent.

 Based on the above properties, it is not immediately apparent that electrostatic ignition of narrow-cut kerosene would be a potential cause of fire in
20 a solvent extraction plant operating with narrow-cut kerosene.

 The research program included a series of solvent ignition trials at the University of Southampton.

25

 The purpose of the trials was to determine the electrostatic ignition properties of narrow-cut kerosene at temperatures likely to occur in a copper solvent extraction process operated by the applicant at Olympic
30 Dam.

 The trials were restricted to the conditions and configurations possible in the copper solvent extraction process at Olympic Dam. These conditions were partially
35 simulated using a 600mm diameter polyethylene pipe, various types of electrostatic discharge including (a) brush, (b) propagating brush, and (c) spark, and various

solvent configurations including aerosol, foam and saturated particulates. During the trials, physical parameters, such as temperature and droplet size distribution (where appropriate), were carefully monitored and the nature of the ignition and subsequent flame propagation throughout the media, when they happened, were examined.

The trials included:

10

(i) Ignition of a solvent-wetted pipe wall as a function of temperature with various electrostatic discharges.

15

(ii) Ignition of a solvent-saturated mineral deposit as a function of temperature with various electrostatic discharges.

20

(iii) Ignition of coarse and fine solvent aerosol from a hydraulic nozzle.

(iv) Ignition of coarse solvent droplets dispersed in a Hartmann tube apparatus.

25

(v) Ignition of dispersed solvent-saturated inert mineral particles sieved in order to control particle size in Hartmann tube apparatus.

30

(vi) Ignition of a foaming solvent on a liquid surface.

The results of the trials and electrostatic measurements on site at Olympic Dam indicated that:

35

(a) high levels of electrostatic charge could be generated with narrow-cut kerosene when transported

through plastic and metal pipes; and

5 (b) the levels of charge generated at even relatively low flow velocities could, under the right conditions, result in electrostatic brush, propagating brush and spark discharges within a copper solvent extraction plant.

10 It was clear from the trials and the work on site at Olympic Dam that the co-existence of electrostatic discharges and particular forms of narrow-cut kerosene, such as foams and mists, creates a potential fire hazard. In particular, the trials and the work on site at Olympic Dam, demonstrated that even relatively low electrostatic
15 discharge energies could result in an ignition which is capable of propagation through narrow-cut kerosene in foam or mist form. Once this condition is reached, the quantity and movement of fuel around a copper solvent extraction plant has the capability of producing rapid
20 spread of a resultant fire.

Preferred conductivity modifiers are reagents sold under the trade marks Stadis 425, Stadis 450, Octastat 2000 and Octastat 3000, all supplied by Octel
25 Performance Chemicals.

Stadis 425 is 10-20% toluene, 60-70% kerosene, and 2-7% solvent naphtha, and 2-8% DBSA (dodecylbenzenesulphonic acid)
30

Octastat 2000 is 10-20% toluene, 2-8% DBSA, 50-70% kerosene, and 2-7% TS polymer containing S.

Octastat 3000 is 40-50% toluene, 0-5% propan-2-ol, 5-15% DINNSAA (dinonylnaphthasulphonic acid), 15-30% solvent naphtha, 1-10% TS polymer containing N, and 10-20% polymer containing S.
35

Stadis 450 is 40-50% toluene, 15-25% solvent naphtha, 5-15% DINNSA, and 1-5% propan-2-ol

5 The amounts of conductivity modifier required to increase the conductivity of a solvent to reduce the electrostatic discharge hazard of the solvent to obtain an adequate fire safety level in any given situation will depend on the target electrical conductivity of the
10 solvent and the nature of the solvent being modified.

 In addition, the ionic strengths or composition of the aqueous solution that comes into contact with a solvent in a solvent extraction process will also affect
15 the amount of modifier required to be added to increase the conductivity of the solvent to reduce the electrostatic discharge hazard of the solvent to obtain an adequate fire safety level.

20 In the particular case of a copper solvent extraction process, preferably the solvent is a narrow-cut kerosene and the copper extractant is an oxime which is dissolved in the narrow-cut kerosene solvent.

25 In the above-described particular case, preferably the amount of oxime in the narrow-cut kerosene is between 5-25% by volume of the total volume of oxime and narrow cut kerosene.

30 It is preferred particularly that the amount of oxime in the narrow cut kerosene be between 5-15% by volume of the total volume of oxime and narrow-cut kerosene.

35 In order to reduce the electrostatic discharge hazard of a solvent to obtain an adequate fire safety level, it is preferred that the electrical conductivity of

the solvent in the solvent extraction process be maintained at or above 240 pS/m.

5 More preferably the electrical conductivity of the solvent in the solvent extraction process is maintained at or above 350 pS/m.

10 More preferably the electrical conductivity of the solvent in the solvent extraction process is maintained at or above 450 pS/m.

15 It is preferred particularly that the electrical conductivity of the solvent in the solvent extraction process be maintained at 500 pS/m.

20 In the case of Stadis 425 conductivity modifier, a minimum concentration of 7.5 $\mu\text{L/L}$ gives a conductivity of modified copper solvent of 240 pS/m. For Stadis 450 conductivity modifier, a minimum concentration of 5.0 $\mu\text{L/L}$ gives the requisite conductivity of 260 pS/m. For Octastat 2000 conductivity modifier, a minimum concentration of 7.5 $\mu\text{L/L}$ gives the requisite conductivity of 240 pS/m, and for Octastat 3000 conductivity modifier a minimum concentration of 5.0 $\mu\text{L/L}$ gives a conductivity of 360 pS/m. All of these conductivities are on or above a desired conductivity of 240 pS/m to achieve adequate safety levels.

30 Surprisingly, and advantageously, the use of the above-described conductivity modifier to increase the conductivity of the organic solvent used in a copper solvent extraction process does not adversely impact on the performance of the solvent in the process.

35 The time normally taken for phase separation between aqueous and solvent phases in a solvent extraction process is one measure of process performance. Phase

separation takes place after a metal such as copper is extracted from an aqueous phase into an organic solvent and usually occurs in large settler tanks. The time required for phase separation impacts on the cost of the process. The applicant found in the research program that phase separation time was not unduly affected by the addition of a conductivity modifier to the solvent.

The performance of the extractant used in a solvent extraction process is another measure of the performance of the process. The applicant found in the research program that extractant performance did not appear to be influenced by the addition of a conductivity modifier to the solvent.

The research program included the following laboratory bench trials, described as Examples 1 and 2, that demonstrate the effect of adding conductivity modifiers to an organic solvent used in the copper solvent extraction process operated at Olympic Dam.

Laboratory Bench Trials

Example 1

Four conductivity modifier reagents were tested on plant solvent and pregnant liquor to assess their impact on conductivity and phase separation.

Plant samples from a copper solvent extraction plant were collected in new glass bottles that had been cleaned first with hot water, then with demineralised water, and finally with heptane. No effort was made to remove entrained aqueous phase since entrainment is part of the "reality" of plant solvent.

Test samples consisting of either fresh or plant

solvent containing conductivity modifier reagents were prepared on a mass basis in glass bottles cleaned as previously stated.

5 Four conductivity modifier reagents were tested, namely: Stadis 425, Stadis 450, Octastat 2000, and Octastat 3000.

10 For each conductivity modifier reagent, 5 mL of the reagent was diluted to 500 mL (410.5 g) giving 10000 μ L of conductivity modifier reagent per L of stock solution. This was then diluted 20 mL to 500 mL (410.5 g) giving 400 μ L/L stock solution. This was subsequently diluted 5, 10, 15 and 20 mL to 800 mL (656.8 g) giving 15 2.5, 5.0, 7.5 and 10.0 μ L/L test solutions. Stripped solvent from the plant was used in all dilutions.

Electrical conductivity of each test solution was measured using liquid conductivity meter model L30 20 supplied by the Department of Electrical Engineering, University of Southampton.

Phase separation times were determined by measuring 400g pregnant liquor solution ("PLS") and 328.4 25 g (400 mL) solvent into a baffled one litre beaker. Beaker markings were used to place the agitator in a similar position for each test. After agitation at 300 rpm for 2 minutes the time for the phase separation to reach 200 mL, 300 mL and 350 mL for each sample was recorded.

30

Results

Unmodified solvent had a conductivity of 40 pS/m, while conductivity data for modified solvent is shown in 35 Table 1.

Table 1. Conductivity (pS/m) of modified copper solvent

at various concentrations.

Reagent	Reagent Concentration			
	2.5 µL/L	5.0 µL/L	7.5 µL/L	10.0 µL/L
Stadis 425	100	140	240	300
Stadis 450	130	260	410	590
Octastat 2000	80	150	240	320
Octastat 3000	170	360	550	720

5 In terms of conductivity improvement, it is apparent that Octastat 3000 conductivity modifier was significantly better than any of the other modifiers.

10 Phase separation measurements are set out in Table 2.

Table 2. Phase separation times (minutes) for various mixtures (S=Stadis, O=Octastat)

Separation	Unmodified Solvent			10 µL/L S425	10 µL/L S450	10 µL/L O2000	7.5 µL/L O3000	10 µL/L O3000
200 mL	20	18	20	20	20	19	20	21
300 mL	33	32	36	37	37	35	34	35
350 mL	-	53	55	55	56	52	51	55

15 It is evident from Table 2 that there was no statistical difference in phase separation between samples without conductivity modifiers and samples with conductivity modifiers at concentrations targeting 500
20 pS/m conductivity.

Conclusions

The above results indicate that conductivity modifiers had very little effect on phase separation.

Example 2

5

Two conductivity modifier reagents were added at various concentrations to plant solvent (Shellsol™ narrow-cut kerosene) and copper extractants (Acorga oxime or LIX oxime). The resultant solutions were loaded and stripped with pregnant liquor to assess the impact of these process steps on conductivity and phase separation.

10

Method

15

The method of preparing solutions containing plant solvent and standard additions of conductivity modifier reagent was essentially the same as in Example 1, except that fresh Shellsol narrow-cut kerosene was used in all dilutions, and the samples were prepared on a volume basis (using volumetric flasks) rather than on a mass basis.

20

Anything in contact with solvent was cleaned using hot water, demineralised water, and then heptane. Cleanliness was checked by measuring the conductivity of the final wash of heptane, which had to be less than 5 pS/m.

25

A bulk Acorga oxime solution containing 10% v/v Acorga oxime in fresh Shellsol narrow-cut kerosene was prepared and then conditioned by shaking with strong electrolyte at a ratio of 2.5:1 and then discarding the electrolyte.

30

A bulk LIX oxime solution containing 10% v/v LIX oxime in fresh Shellsol narrow-cut kerosene was prepared

and then conditioned by shaking with strong electrolyte at a ratio of 2.5:1 and then discarding the electrolyte.

For each conductivity modifier reagent (Stadis 450 and Octastat 3000), 5 mL was diluted to 100 mL giving 50000 µL conductivity modifier reagent per L of stock solution. This was then diluted 5 mL to 250 mL giving 1000 µL/L stock solution. This was subsequently diluted: (a) 5, 10, 15 and 20 µL to 1 litre of plant solvent, (b) 5, 10, 15 and 20 µL to 1 litre of fresh 10 % Acorga oxime solution, and (c) 5, 10, 15 and 20 µL to 1 litre of fresh 10 % LIX solution, giving 5, 10, 15 and 20 µL/L test solutions.

In each test run, a 2000 mL beaker with baffles was loaded with 1000 mL PLS and 500 mL test solution. The mixture was agitated for 5 minutes, and the time taken for separation to a mark on the beaker just below 1000 mL was recorded.

After loading, the entire contents were transferred to a 2 L separation funnel and the raffinate was discarded after collection of sample for analysis. Conductivity of a portion of the test solution was measured, and 400 mL collected for stripping using a measuring cylinder. Remaining test solution was used for analysis.

The test solution was transferred to a 1 L glass bottle, and 160 mL weak electrolyte added. An agitator with hinged blades was inserted into the bottle and the concoction was then mixed at 400 rpm for 5 minutes. Separation times were initially recorded, but the reliability and usefulness was very poor because bubble formation around the interface made it very difficult to get reproducible times.

For the test involving multiple loading/stripping, exactly the same procedure was used, but because of solvent lost through sample collection and

entrainment, replicate loading/stripping tests were combined at each stage so that there would be sufficient test solution left for the final load/strip. The sequence is shown in Table 3.

5.

Table 3. Loading and stripping volumes for multi-stage extractions.

Cycle	Load	Strip
1	5 x 500 mL = 2500 mL	5 x 400 mL = 2000 mL
2	3 x 500 mL = 1500 mL	3 x 400 mL = 1200 mL
3	2 x 500 mL = 1000 mL	2 x 400 mL = 800 mL
4	1 x 500 mL = 500 mL	1 x 400 mL = 400 mL

10 Results

Tables 4 and 5 present electrical conductivity and phase separation times for loaded and stripped test solutions containing added modifiers. Table 6 highlights the change in electrical conductivity as test solutions were loaded and stripped a number of times.

Table 4. Conductivity (nS/m) of copper solvent from various sources with added conductivity.

Solvent Source	Modifier Concentration $\mu\text{L/L}$	Octastat 3000 Conductivity (nS/m)			Stadis 450 Conductivity (nS/m)		
		Start	Loaded	Stripped	Start	Loaded	Stripped
Plant Stripped Solvent	0	0.08	0.03	0.02	-	-	-
	5	0.53	0.52	0.53	0.39	0.41	0.32
	10	1.07	1.06	1.18	0.88	0.82	0.77
	15	2.00	2.32	N/A	1.72	1.62	1.12
	20	3.03	2.88	1.55	2.72	2.26	1.26
10% v/v Acorga in Shellisol	0	0.04	N/A	N/A	-	-	-
	5	0.46	0.38	0.32	0.30	0.50	0.52
	10	0.86	0.76	0.65	0.64	0.89	0.67
	15	1.19	1.14	1.25	0.94	1.71	0.90
	20	1.69	2.49	1.96	1.21	2.55	1.20
10% v/v LIX in Shellisol	0	0.13	0.04	0.10	-	-	-
	5	0.69	0.56	0.81	0.53	0.48	0.47
	10	1.00	1.02	1.21	0.62	0.91	0.89
	15	1.41	1.65	1.77	1.17	1.43	1.24
	20	1.93	2.63	2.17	1.59	1.72	1.77

Table 5. Phase separation times for various mixtures.

Solvent Source	Modifier Concentration ($\mu\text{L/L}$)	Octastat 3000 Separation Time(s)		Stadis 450 Separation Time(s)	
		Loading	Stripping	Loading	Stripping
Plant stripped Solvent	0	60	67	-	-
	5	65	75	68	75
	10	66	78	70	51
	15	76	N/A	76	68
	20	71	94	71	90
10% v/v Acorga in Shellsol	0	N/A	N/A	-	-
	5	60	115	68	75
	10	68	115	68	95
	15	72	105	75	100
	20	77	95	48	N/A
10% v/v LIX in Shellsol	0	46	N/A	-	-
	5	51	N/A	48	N/A
	10	49	N/A	46	N/A
	15	47	N/A	55	N/A
	20	48	N/A	50	N/A

Table 6. Conductivity of 16 μ L/L Octastat 3000 in plant solvent.

Cycle	Conductivity (nS/m)		
	Start	Load	Strip
1	1.80	1.18	0.96
2	-	0.99	0.98
3	-	0.95	0.96
4	-	0.89	0.76

5 Conclusions

10 In terms of electrical conductivity enhancement, Octastat 3000 performed better than Stadis 450 by about 20 to 30%. In addition, multiple loading and stripping of the test solutions resulted in a decrease in conductivity at an apparently modest rate after an initial drop in conductivity.

15 Mini-Plant Trials

In addition to the above laboratory bench trails, the research program included a mini-plant continuous trial carried out by ANSTO.

20 The purpose of the trial was to test the impact of conductivity modifier addition on plant performance.

25 The mini-pilot plant circuit was set up to simulate as closely as possible operating conditions in the copper solvent extraction plant at Olympic Dam.

Two circuits, CIRCUIT 1 (C1) and CIRCUIT 2 (C2), with identical configurations, were operated in parallel.

30 Each circuit consisted of 2 extraction stages, 1 scrub stage and 2 strip stages. The aqueous feed solutions were heated prior to entering the circuits via

glass coils immersed in a water bath. A schematic representation of the set-up is shown in Figure 1.

5 The details of operating conditions are summarised in Table 7 below. The conductivity modifier reagent used for this work was Octastat 3000. It was added to the circuit as a 5000 $\mu\text{L/L}$ solution diluted in Shellsol 2046 natural cut kerosene.

10 Table 1. Summary of Mini-Plant Operating Conditions

		RUN 1 Modifier 20-144 h	RUN 1 Modifier & Clay Treatment 144-240 h
O:A flows	Extraction	1.0	1.0
	Scrub	17	16
	Strip	6.4	3.9
Mixer Retention (min.)	Extraction	1.6	1.7
	Scrub	1.3	1.3
	Strip	1.2	1.3
Settler Load* ($\text{m}^3/\text{h}/\text{m}^2$)	Extraction	4.2	4.0
	Scrub	4.2	4.0
	Strip	4.3	4.0
Temperature		45-31	47-35

* Mini-plant settler loads calculated using barriers to reduced effective settler size to $\frac{1}{4}$ of its total size

15

The mini-plant was operated for 240 hours.

CIRCUIT 1 was the control circuit, CIRCUIT 2 was the test circuit, with addition of conductivity modifier reagent to a solvent reservoir. After 144 h, clay treatment was introduced in both the control and the test circuits.

25 Electrical conductivity, phase separation times and other measurements were made during the operation of

the mini-plant.

The objective of conductivity modifier addition to the mini-plant circuit was to increase the conductivity of the solvent in the circuit to a target of 500 pS/m. This target level had been determined from laboratory bench trials to be sufficient to prevent a build-up and discharge of static electricity, and therefore significantly contribute to reducing the risk of a fire.

The two circuits were set up with solvent being pumped from a reservoir to the extraction circuit, and stripped solvent being returned to the reservoir. Frequent samples were taken from the reservoir and the conductivity measured with a liquid conductivity meter (Wolfson Electrostatics, Model 30). Periodically, solvent samples were also taken from the settlers of the extraction, scrub and strip circuits. All samples were returned to the circuit.

Baseline electrical conductivity data was obtained by measurements of solvent samples taken from CIRCUIT 1 (the control circuit) operated without any modifier. The results indicated that, on average, the conductivity of the solvent reservoir in the control circuit was 35 pS m⁻¹, with similar values measured in the strip circuit. The readings of samples taken from the extraction and scrub circuits were higher than that of the reservoir, with maximum readings of 83 and 101 pS m⁻¹ measured for the two circuits, respectively.

The electrical conductivity of the reservoir of the test circuit, CIRCUIT 2, was also similarly monitored. Addition of small volumes of conductivity modifier (0.2-1 mL at a time) was made to the reservoir to aim for a target conductivity of 500 pS m⁻¹. A stock of 5000 µL/L of modifier in Shellsol 2046 narrow-cut kerosene was used for this purpose. The stock solution was kept in the dark, when not in use. Conductivity modifier was added to

CIRCUIT 2 throughout RUN 1. For RUN 2, modifier addition to CIRCUIT 2 only commenced 48 hours after the start of the run. Conductivity measurements of samples taken from the reservoir extraction, scrub and strip circuits are shown in Figure 2.

The conductivity measurements consistently showed higher values for extraction, and even higher values for scrub solvent samples. The conductivity of the solvent in the strip circuit was similar to that of the reservoir. This observation was consistent for both RUN 1 and RUN 2. In RUN 1, introduction of clay treatment increased the difference between reservoir and scrub solvent conductivities with readings as high as 2000 pS m^{-1} registered. In RUN 2, where there was no clay treatment, conductivity values for scrub varied between $1000\text{--}1600 \text{ pS m}^{-1}$.

Conclusions

- No major differences in phase disengagement characteristics were detected between operation with and without modifier Octastat 3000 doped to a target conductivity of 500 pS/m in the solvent reservoir.

- The presence of conductivity modifier did not cause any increase in the measured organic entrainment levels in both the raffinate and strong electrolyte solutions. The amount of organic entrainment averaged between $25\text{--}140 \text{ ppm}$ in the raffinate and between $19\text{--}28 \text{ ppm}$ in the strong electrolyte.

- The presence of conductivity modifier did not cause any increase in aqueous entrainment in the loaded organic, which averaged at 0.05% .

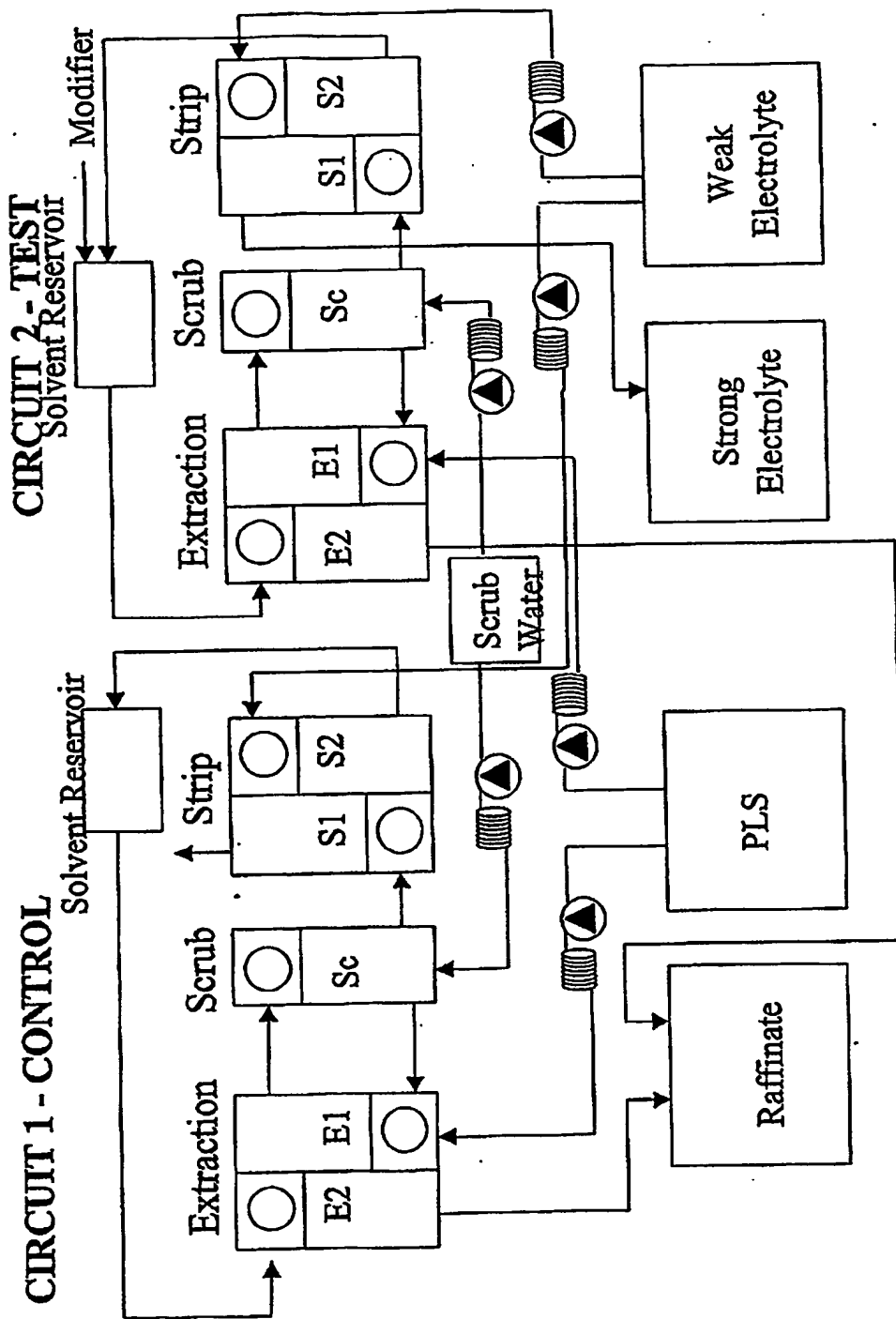
- The presence of conductivity modifier did not increase the amount of impurity carry-over to the strong electrolyte.

• The measured plant data showed that addition of conductivity modifier increased copper extraction. The increase was quite significant (~12%) from a baseline of 55-59%.

• The presence of conductivity modifier consistently resulted in higher levels of conductivity in the scrub and extraction circuits compared to the strip circuit and solvent reservoir. This increase could not be attributed to aqueous entrainment in the solvent or the formation of stable emulsions.

The overall assessment of the mini-plant trial is that addition of Octastat 3000 to a target conductivity of 500 pS/m did not have a short term negative impact on copper solvent extraction and, moreover, caused a significant increase in copper extraction.

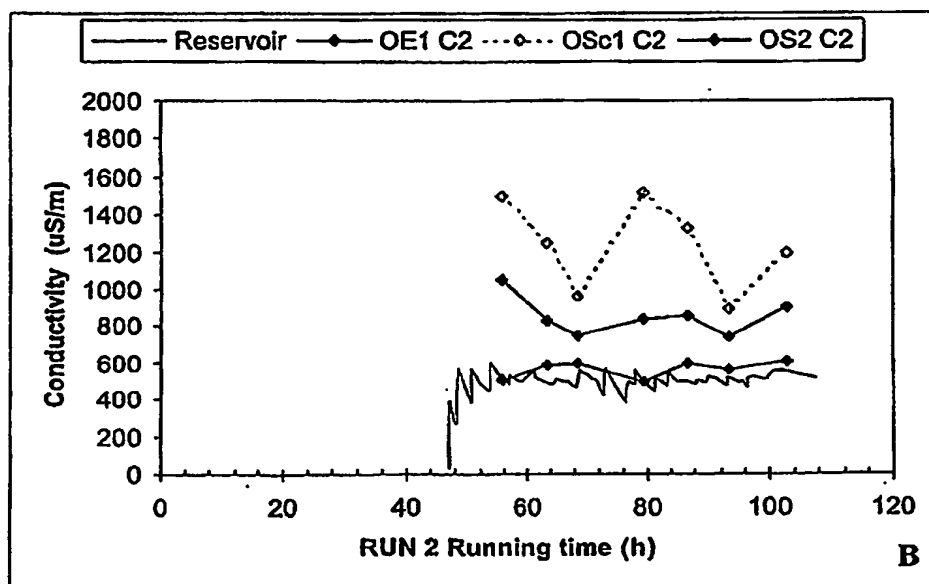
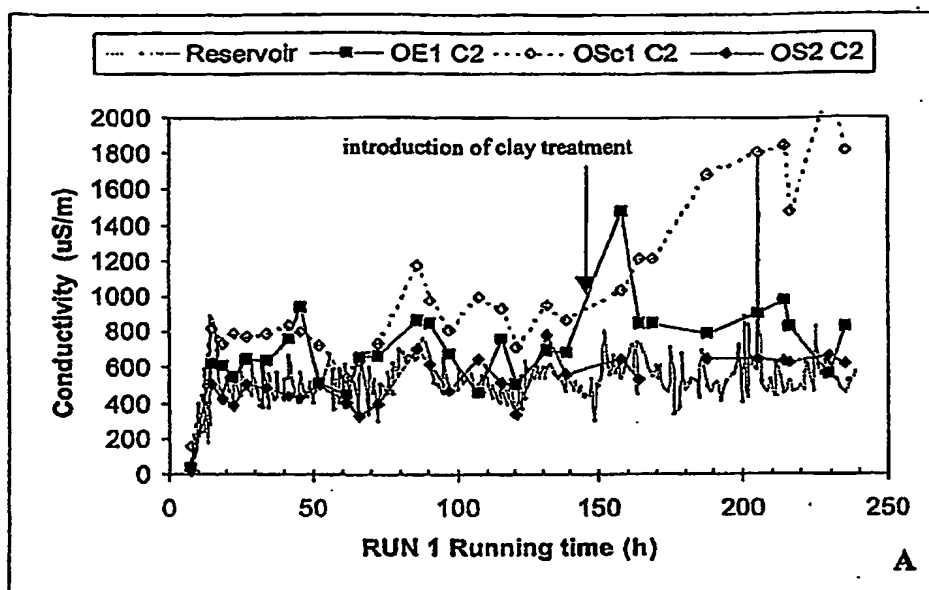
Many modifications may be made to the embodiments of the present invention described above without departing from the spirit and scope of the invention.



Schematic diagram of mini-pilot plant setup

FIGURE 1

FIGURE 2



Solvent conductivity measurements ($\mu\text{S m}^{-1}$) in CIRCUIT 2, A-RUN 1 B-RUN 2